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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.: 10/066,850

Applicant: Ilya AVRUTOV et al.

Filed: February 4, 2002

Art Unit 1625

Examiner: ROBINSON, BINTA M.

For: PROCESS FOR THE PRODUCTION OF SUBSTITUTED 2-(2-PYRIDYLMETHYL)
SULFINYL-IH-BENZIMIDAZOLES

Docket No.: 1662/55702

Customer No.: 23838

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

STATEMENT OF SUBSTANCE OF INTERVIEW UNDER 37 C.F.R § 1.133(b)

SIR:

The undersigned would like to thank Examiner Robinson, Supervisory Primary Examiner McKane and Primary Examiner Chang for a courteous and helpful personal interview conducted on June 22, 2004.

In the interview, the undersigned requested reconsideration of the indefiniteness rejection of claims 16-18, 21, 51 and 52 over the use of the trade name "OXONE." The undersigned argued that the use of "OXONE" in these claims comply with the requirement of MPEP 608.01(v) because the meaning of "OXONE" was well-known in the United States and satisfactorily defined in the literature. Applicants provided the examiners copies of the definition of "OXONE" taken from the Condensed Chemical Dictionary, 1966, to prove that the meaning of "OXONE" is well-known and satisfactorily defined. A copy of the same dictionary definition was attached to the Amendment filed in June 2004. Applicants pointed out that the same definition was disclosed in page 6 of the specification, which also gives DuPont as the source for OXONE.

In addition, during the interview, applicants' counsel provided the examiners with copies of a description of OXONE taken from the Web site of DuPont at www.dupont.com/oxone/techinfo. Per the request of Supervisory Primary Examiner McKane, a copy of the description of OXONE taken from the Web site of DuPont is attached in order to make it of record. No agreement was reached regarding this issue.

Furthermore, applicants' counsel argued that claims 25-28 should not have been rejected as indefinite over the recitation of "sulfone." Applicants took a position that it was well known that compounds prepared synthetically could contain by-products, so it would not be indefinite to claim omeprazole, lansoprazole, etc., having less than a certain amount of a sulfone by-product. The examiners did not voice any disagreement concerning applicants' argument that these claims were not indefinite.

Respectfully submitted,

KENYON & KENYON

Dated: July 22, 2004

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Enclosure
495653_1



Oxone® monopersulfate compound

DuPont Oxone®

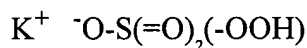
Monopersulfate Compound Technical Information

- [Chemistry and Product Properties](#)
- [Solutions](#)
- [Safety and Handling](#)
- [Storage Conditions](#)
- [Packaging and Shipping](#)
- [Processing of Oxone®](#)
- [Test Methods](#)

Chemistry and Product Properties

Active Ingredient

The active ingredient of Oxone® is potassium peroxymonosulfate, KHSO_5 [CAS-RN 10058-23-8], commonly known as potassium monopersulfate, which is present as a component of a triple salt with the formula $2\text{KHSO}_5 \cdot \text{KHSO}_4 \cdot \text{K}_2\text{SO}_4$ [potassium hydrogen peroxymonosulfate sulfate (5:3:2:2), [CAS-RN 70693-62-8]. The oxidation potential of Oxone® is derived from its peracid chemistry; it is the first neutralization salt of peroxymonosulfuric acid H_2SO_5 (also known as Caro's acid).



Potassium Monopersulfate

Oxidation Potential

The standard electrode potential (E°) of Oxone® is shown in the following reaction:



This potential is high enough for many room temperature oxidations, including halide to halogen or hypohalite, ferrous ion to ferric, and manganous ion to manganic.

The physical properties and typical analyses of Oxone® are shown in Table I.

TABLE I

DuPont Oxone® Physical Properties and Typical Analysis

Molecular Weight (triple salt)	614.7
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Active Oxygen	% min.	4.5
	% average analysis	4.7
	% theoretical (triple salt)	5.2
Active Component	% KHSO ₅ , min.	42.8
Bulk Density	g/cm ³ (Mg/m ³)	1.15-1.30
	lb/ft ³	72-81
Particle Size	through USS Sieve #20, %	100
	through US Sieve #200, % min.	10
	(also see Table II)	
pH	at 25°C (77°F)	
	1% solution	2.3
	3% solution	2.0
Solubility	g/L, H ₂ O, 20°C (68°F)	256
	(also see Table III)	
Moisture Content	% max.	0.1
Stability	% active oxygen loss/month	<1
Standard Electrode Potential (E°)	V	-1.44
Heat of Decomposition	kJ/kg	251
	Btu/lb	108
Thermal Conductivity	W/m K	0.161
	Btu ft/h ft ² F	0.093

This Table shows typical properties based on historical production performance. DuPont does not make any express or implied warranty that this product will continue to have these typical properties.

Stability

Oxone® is a relatively stable peroxygen, and loses less than 1% of its activity per month when stored under appropriate conditions. However, like all other peroxygens, Oxone® undergoes very slow decomposition in storage, with liberation of oxygen gas and a small amount of heat. Decomposition of Oxone® generates oxygen gas. If a decomposition is associated with high temperature, decomposition of the constituent salts of Oxone® may generate sulfuric acid, sulfur dioxide, or sulfur trioxide. The stability is reduced by the presence of small amounts of moisture, alkaline chemicals, chemicals which contain water of hydration, transition metals in any form, and/or any material with which Oxone® can react. The decomposition of Oxone® is exothermic; this property can cause the decomposition to accelerate if conditions allow the product temperature to rise.

Particle Size Distribution

The typical particle size analysis of Oxone® is shown in Table II. Particle size may be adjusted by screening, grinding, or compaction/granulation processes. The product temperature should be kept below 50°C (122°F) at all times during such operations; packaging temperature should not exceed 30°C (86°F).

TABLE II
Typical Particle Size Analysis of Oxone® Monopersulfate

U.S. Sieve Size	Sieve Opening, μm	Approx. Composition, wt% (On Screen, Cumulative)
30	600	1
70	212	68
100	150	84
200	75	98
325	45	100

Solutions

Oxone® is highly and readily soluble in water as shown in Table III. At 20°C (68°F), the solubility of Oxone® in water is >250 g/L.

At concentrations above saturation, potassium sulfate will precipitate, but additional potassium monopersulfate can remain in solution, so that the attainable % active oxygen in solution is higher than is indicated in Table III.

Solutions of Oxone® are relatively stable when made up at the unmodified pH of the product. The stability is adversely affected by higher pH, especially above pH 7. A point of minimum stability exists at pH 9, at which the concentration of the mono-anion HSO_5^- is equal to that of the di-anion $\text{SO}_5^{=}$.

Iron, cobalt, nickel, copper, manganese, and other transition metal ions can catalyze the decomposition of Oxone® in solution; the degree to which catalysis occurs is dependent on the concentrations of Oxone® and of the metal ion.

Solubility of Oxone®

TABLE III
Solubility of Oxone® Monopersulfate in Water

Temperature, °C °F	Solubility, g/L	Active Oxygen In Solution, wt%
20 68	256	0.92
27 80	268	0.95
49 120	300	1.04

60	140	315	1.08
71	160	335	1.13

Storage of Oxone® Solutions

Solutions of Oxone® can be prepared readily because of its rapid and high solubility and are reasonably stable for up to a few weeks if high quality water is used and if temperatures are kept low [preferably <25°C (77°F)]. Solutions should be stored in vessels of appropriate materials of construction such as stainless steel or polyethylene. Vessels should be loosely covered to avoid airborne contaminants, but should be vented to prevent pressure buildup. Upward adjustment of the pH, any addition of a reactive or catalytic material, or an increase in temperature will decrease the stability of an Oxone® solution.

Such solutions can be metered to a process as required. Equipment for dissolution of Oxone® from 25-kg bags or 1-t (metric) bulk bags is available from DuPont; for further information see your Sales Representative or call 888-243-4608 (302-892-7575 outside the U.S. and Canada).

Safety and Handling

Personal Safety and First Aid

The Oxone® Material Safety Data Sheet (MSDS) provides additional information about the product, and should be consulted before Oxone® is handled. Ask your Sales Representative or call 800-441-9340 for a copy of the MSDS.

Health Hazards

Oxone® has a low order of toxicity when taken internally, based on animal studies. The approximate lethal dose (ALD) for rats is 2250 mg/kg. However, Oxone® should not be taken internally and should be considered harmful if swallowed.

Oxone® is corrosive to the eyes, skin, nose, and throat due to its acidity and oxidizing properties, and may cause allergic reactions in sensitive individuals. Temporary hair loss has been observed on contact with moist or sweaty skin. DuPont observes an airborne exposure limit to Oxone® dust of 1 mg/m³, 8-hour time-weighted average.

Safety Precautions

Persons handling Oxone® should avoid contact with eyes, skin, or clothing. Avoid breathing dust. Wash thoroughly after handling and launder contaminated clothing before re-use. Exposure to Oxone® can be minimized by providing adequate ventilation and by wearing rubber- or plastic-coated gloves and chemical safety goggles.

Site Facilities

The following safety equipment should be easily accessible in all areas where Oxone® is handled or stored:

- Safety Showers with quick opening valves that stay open. Water should be supplied through insulated lines.
- Water Hydrant and Hose or other means of flushing spills with large volumes of water under low pressure.
- Eye Wash Fountains or other means for thoroughly washing the eyes with a gentle flow of clean water.

First Aid

In case of contact, immediately flush eyes with plenty of water for at least 15 min. Flush skin with water. If inhaled, remove to fresh air. Call a physician.

Storage Conditions

Oxone® should be stored in cool, dry areas, away from combustible materials, incompatible chemicals, and sources of heat such as space heaters and light fixtures. (see Incompatible Materials). Prolonged storage at ambient temperatures greater than 32°C (90°F) should be avoided. Pallets of 25-kg bags can be stacked if there are 2-3 in of air space between them; however, pallets should be stacked no more than two (2) high unless local fire codes permit higher stacking. Torn bags and product spillage must be thoroughly cleaned up and removed from the area (see Waste Disposal).

Oxone® decomposition will be accelerated on contact with moisture. Product packaging includes a water-resistant liner, but storage conditions should also include provisions for prevention of contact with water, including high airborne humidity.

Mass Limitation

Like all other peroxygens, Oxone® undergoes very slow decomposition which liberates heat (heat of decomposition = 251 kJ/kg). The maximum mass of Oxone® during storage or processing at ambient temperature should never be allowed to exceed a cube 122 cm (4 ft) on each side; this is equivalent to 1.81 m³ (64 ft³) or 2045 kg (4500 lb) of Oxone®. Quantities in excess of this limit can trap heat or prevent heat dissipation, which can lead to runaway decomposition with liberation of large amounts of oxygen, additional heat, and eventually to acidic fumes containing SO₂ or SO₃ if the internal mass temperature exceeds 300°C (572°F). Storage silos, hoppers, processing vessels, blenders, and other facilities must be designed to avoid quantities in excess of this limitation.

Blending Oxone® with other materials usually reduces the safe storage mass.

Incompatible Materials

Incompatible materials are those which can cause accelerated decomposition of Oxone® or which can react with Oxone® to form undesirable products. While Oxone® might co-exist with such materials under ideal conditions, they must be avoided to prevent serious consequences should unanticipated conditions occur (for example, inadvertent contact with moisture).

The following are considered incompatible materials and should not be transported or stored in proximity to Oxone®:

- Compounds containing halides or active halogens. Oxone® can oxidize halides to active halogens (for example, chloride to chlorine), and the acidity of Oxone® might react with an active halogen compound to release halogen gas.
- Cyanides, which can react with the acidity of Oxone® to release toxic hydrogen cyanide gas.
- Transition "heavy" metals (such as copper, manganese, cobalt, or nickel) or their salts, oxides, hydroxides, etc., can accelerate the decomposition of Oxone® with evolution of oxygen gas.
- Readily oxidizable organic compounds.

Waste Disposal

Spillage, floor sweepings, and other waste Oxone® should be dissolved, diluted and disposed of in accordance with Federal, State, and local regulations. Solutions of greater than 3% Oxone® will have a pH less than 2.0, and may be considered RCRA hazardous due to the low pH. Neutralization with caustic soda or soda ash may be necessary before flushing to waste treatment plant or sewer, if approved by authorities.

Spilled material must not be recycled to production.

Solid Oxone® waste, including dust from dust collectors or bag houses, must be collected in separate containers and kept segregated from other materials.

Packaging and Shipping

Shipping Containers

Oxone® is available in 25-kg (55.1-lb) net multi-wall, moisture-resistant paper bags (42 per pallet), 12-kg repulpable bags (80 per box), and 1-t (metric) (2205 lb) lined bulk bags.

Formulated mixtures containing Oxone® should always be protected by moisture barrier packaging.

Shipping Information

Proper Shipping Name = CORROSIVE, SOLID, ACIDIC, INORGANIC, N.O.S.

(MONOPERSULFATE COMPOUND)

Hazard Class = 8

UN Number = 3260

DOT/IMO Label = CORROSIVE

Packing Group = 2

Other jurisdictions may have additional requirements and designations.

Processing of Oxone®

Persons handling Oxone® should avoid contact with it (see [Safety Precautions](#)).

Materials of Construction

The primary consideration in choosing equipment for processing Oxone® and solutions of Oxone® is prevention of contamination of the product or reaction with the equipment. Stainless steel, porcelain, polyethylene, and Teflon® are generally suitable. Iron, steel, copper, brass, and other materials which could introduce decomposition catalysts must not be used.

Conditions for Processing

Dedicated equipment should be used for processing Oxone® whenever possible; otherwise, thorough cleaning of the equipment is needed to remove all other materials from it. Equipment must be dried completely; any introduction of moisture into Oxone® must be avoided.

Spillage of Oxone®, and dust collected in bag houses or filters during processing, should be handled as waste (See [Waste Disposal](#)) rather than recycled unless conditions prevent contamination or moisture pickup.

Hoppers, feeders, blenders, and other processing equipment should be sized to prevent exceeding the mass limitation of Oxone® (see [Mass Limitation](#)).

Since Oxone® can absorb moisture from the air, the relative humidity should be kept as low as possible during processing. Oxone® will not absorb moisture at R.H. < 50% if the ambient temperature is kept below 36°C (97°F); however, blends may absorb moisture more readily. Some operations use 15% R.H. to produce long-term stable blended products containing Oxone®.

Blending Oxone®

Blends of Oxone® must be made with anhydrous ingredients and conditions should be set to minimize moisture pickup. Blending equipment should be clean and dry. Clearances should be set so that abrasion or grinding of Oxone® particles is minimized. If screw feeders are used, they should be slow turning in order to minimize heat generation and buildup of Oxone® on machine surfaces.

Upon completion of blending, the mixture should be cooled to < 30°C (86°F) and packaged immediately into moisture-proof containers to avoid moisture pickup from the air. Temporary in-process storage of blended materials should be avoided. The effect of blending and packaging variables on the stability of a blend should be determined before commercial scale operations are begun.

Any temperature rise during blending may indicate the onset of decomposition and should be immediately investigated.

Oxone® can be blended with a wide variety of additives, including sodium sulfate, sodium carbonate (especially dense), sodium bicarbonate, sodium sesquicarbonate, sodium perborate (monohydrate or anhydrous, but not tetrahydrate), tetrasodium pyrophosphate, sodium tripolyphosphate, sodium

metasilicate; citric, malic, and tartaric acids, wetting agents and detergents, and fragrances. All ingredients used with Oxone® should be anhydrous or should hold hydrated water tightly; stability testing of each blend is essential.

Since Oxone® is acidic, it is usually blended with sufficient alkaline salts to buffer it to near-neutral or slightly alkaline pH when compounded into cleaners.

Formulations of Oxone® containing chloride ions can be used to generate low concentrations of active chlorine in cleaning systems, provided the blend is pH buffered to avoid evolution of chlorine gas, and special care is taken to exclude moisture from the formulation; bromides and iodides are more reactive and generally should not be blended with Oxone®.

CAUTION: Such formulas must include sufficient alkali to avoid generation of elemental chlorine, a highly toxic gas.

Some organic compounds such as anionic or non-ionic detergents, chelating agents, dyes, and optical brighteners, flavors, and fragrances can be used as minor components of blends containing Oxone®; such blends should be carefully evaluated for stability before use.

Compaction and Granulation

The particle size distribution of Oxone® can be increased by compacting, granulating, and screening. This procedure is often used before tableting, but may also be of interest for any application in which the minimum particle size is specified.

Test Methods

Active Oxygen/Active Component

1. Take a representative sample by riffing, quartering, blending, or other means.
2. From the sample, carefully weigh at least two specimens of 0.4 +/- 0.001 g. each.
3. Add to a 250-mL beaker equipped with magnetic stirrer: 75 mL deionized water, 15 mL 10% (v/v) sulfuric acid, and 10 mL 25% (w/w) potassium iodide solution. (Deionized water and all reagents should be < 20°C). Add a weighed specimen of Oxone® and stir until dissolved.
4. Immediately titrate the specimen with 0.1 N sodium thiosulfate solution to a pale yellow color. Add 2 mL starch indicator solution, and the solution will turn deep blue. Immediately continue the titration to a colorless endpoint that persists for at least 30 sec.
5. Calculations
$$\% \text{ active oxygen} = (\text{mLthio} \cdot N_{\text{thio}} \cdot 0.008 \cdot 100) / \text{specimen weight (g)}$$
$$\% \text{ active component (KHSO}_5\text{)} = \% \text{ active oxygen} / 0.1053$$
6. Report the average of specimens analyzed.

Moisture Content

1. Using the sampling procedure described above, weigh at least two 10 +/- 0.01 g specimens in tared weighing dishes.
2. Dry for 30 min in an oven at 65 +/- 0.5°C (149 +/- 1.0°F).
3. Remove the specimens to a desiccator and cool to room temperature.
4. Reweigh the specimens.
5. Calculations
$$\% \text{ moisture} = (\text{original weight} - \text{dry weight}) (100) / (\text{original weight})$$
6. Report the average of specimens analyzed.

Other Testing Methods for Oxone®

- Low concentrations of Oxone® (0-20 ppm), which are commonly used in swimming pool treatments, can be measured in the presence of active chlorine by special test kits offered by Taylor (Model K-1518) and Lamotte (Model 3360).
- In the absence of active chlorine, low concentrations of Oxone® can be measured with a standard DPD-4 test kit; the result must be multiplied by 5.0 to obtain the correct Oxone® concentration in ppm.
- In higher concentrations, Oxone® can be measured by addition of a known quantity of ferrous ammonium sulfate (in excess), followed by back-titration with standardized potassium permanganate or ceric sulfate solution.

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If you have questions or comments, please contact us by e-mail; by phone at DuPont Specialty Chemicals: 1-888-243-4608 (302-892-7575 outside the U.S. and Canada); or by fax: 302-892-1456.

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